OVEN METHODS FOR PRECISE MEASUREMENT OF MOISTURE CONTENT OF SEEDS

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U. S. DEPARTMENT OF AGRICULTURE

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PREFACE

This study is part of a broad program of marketing research designed to maintain quality of farm products, develop and expand markets, improve marketing services, and hold down the costs and increase the efficiency of marketing farm products. This phase of the program is conducted to develop techniques, methods, and devices for the objective measurement of quality factors in agricultural commodities.

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SUMMARY

Farmers, seedsmen, and processors are interested in the moisture content of seeds because it is an important factor in the retention of viability and storage life. In this study air-oven procedures have been standardized to determine accurately the moisture content of 32 common agricultural and vegetable seeds. For 23 of these seeds, there has been no generally accepted standard method to determine moisture. The only laboratory instruments required for this procedure are a constant-temperature air oven and an analytical balance. The analyses can be run by any capable laboratory technician.

The time and temperature used for drying each seed in this method were selected by standardization against the Karl Fischer method. Because the Karl Fischer method is based upon a chemical reaction of the water in the seeds, it is free of the errors inherent in the oven methods.

Errors that may be introduced in the oven procedure and by grinding are reviewed. Two other sources of error are illustrated by a series of experiments. Drying seeds to constant weight at different temperatures showed that apparent moisture content determined by oven procedure is dependent upon the temperature of drying. Wheat, corn and flaxseed gave different apparent moisture contents for different drying temperatures. Of 18 vegetable and hay seeds analyzed by the air-oven method specified by the United States Department of Agriculture for cereal grains (1 hour at 130° C.), 14 gave higher moisture results than when analyzed by the Karl Fischer method. This experiment illustrated the type of error that may be introduced by using the same time and temperature of drying for all seeds.

OVEN METHODS FOR PRECISE MEASUREMENT OF MOISTURE IN SEEDS

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INTRODUCTION

Moisture content of seeds is one of the most important factors in the maintenance of seed quality. From time of harvest, through steps of marketing to time of planting, seed moisture varies. The variation is affected by initial moisture content and the environment to which the seed is exposed. If the moisture content rises above certain critical levels for any appreciable time period, there is danger of undesirable stimulation of physiological processes within the seed with consequent weakening and loss of viability. Knowledge of moisture content is needed to decide whether seed should be dried down before storage or shipment and to determine proper conditions of storage.

Methods for measuring moisture content are generally classified into two types: basic and practical. Basic methods measure loss of water upon heating or measure water directly by chemical or other means. Oven methods and the Karl Fischer chemical method are examples. Practical methods are rapid methods specifically designed for routine use. They may be modifications of the basic methods or operate on entirely different principles. Electric moisture meters are examples of the practical methods. Practical methods are standardized against one or more basic methods. Basic methods vary in reliability. Chemical methods are used to calibrate the simpler oven methods (*4*).[1]

The United States Department of Agriculture and other organizations (*1*, *7*, *8*) specify oven methods for determining the moisture content of cereal grains and oilseeds. These official methods have been checked against chemical methods (*4*), but for many other common agricultural seeds there are no generally accepted methods for determining moisture content. This study is designed to fill this need. For reasons of simplicity, low cost, and availability, oven procedures were considered the best choice to develop the desired methods. These oven procedures were checked against a reference method acknowledged to be accurate.

BACKGROUND

Errors in Oven Method

Effects of Heating

The starch in seeds dried in an oven may be dextrinized if they contain acids or acid salts. In this reaction some of the water which would otherwise be considered as moisture combines with the starch (*3*).

Reducing sugars may condense with amino compounds in the familiar browning reaction and give water as a product. The resulting glycosylamine is unstable to heat, particularly as the system is brought to dryness. The result is further decomposition and additional loss of water and carbon dioxide (*3*). When the seeds contain sugar and soluble protein, the possibility of error from this source is especially great.

[1] Italic figures in parentheses refer to items in Literature Cited, page 10.

When seeds contain an oil of high iodine number, the oil is likely to undergo oxidation on heating. This reaction produces an increase in weight and an apparent decrease in moisture content. Grinding of the seed will increase the probability of error from this source.

Effects of Grinding

The official methods set by the United States Department of Agriculture (7) require that all seeds for which standards have been established, except corn, beans, and seeds with high oil content, be ground before oven-drying. Because seeds of different plants vary in shape, size, hardness, gumminess, ratio of seedcoat to endosperm and germ, the size and shape of particles resulting from grinding in the same mill are often different. Because moisture in the seed has an effect on the grinding, seeds of high moisture content are often predried. Seeds may gain or lose moisture during grinding, some are predried to lessen gumminess. Because of the variation introduced by grinding, it seems improbable that moisture would be lost at the same rate from the ground seeds of different plants, or that the same drying time could be used for all seeds. Particles of ground seeds containing glue-like material may, on heating, become encased in a tough surface film which tends to delay the release of moisture from the inside (2).

Suggested Reference Methods

Phosphorus Pentoxide Method

The rules of the International Seed Testing Association (5) specify oven methods that are similar to well-recognized or official methods (1, 7, 8). The rules also list the vacuum phosphorus pentoxide method of Leendertz (6) in which the seeds are dried to constant weight at 80° C. in a vacuum. The moisture is absorbed on phosphorus pentoxide placed in the same tube with the sample. The rules state that this method is very accurate and is relatively free of the following errors: the influence of the humidity of the air upon the sample, incomplete determination of moisture, and decomposition of the sample from high temperature. The International Seed Testing Association recommends the vacuum phosphorus pentoxide method as a control in calibrating other methods suitable for routine work. The use of lower temperature and pressure, however, may not eliminate the possibility of losing nonaqueous volatile matter. Although it is not so stated in the rules of the Association it appears that the criterion for fixing the drying time in each method is the time required to dry to constant weight. It will be shown in this report that in drying some seeds the loss in weight, upon reaching constant weight, is dependent upon the temperature used.

Karl Fischer Method

Determination of the moisture content of seeds by the Karl Fischer method is based upon the chemical reaction of water with Karl Fischer reagent, named for the originator of the method (4). The water is extracted from the seed with a water solvent, methanol, and titrated with the reagent. A modified Stein mill is used for simultaneously grinding and extracting the grain samples.[2] The water is extracted from the grain sample in 5 minutes at a temperature of 64.5° C. Because the grain is ground and extracted quickly, in a closed cup at low temperature there is little chance for moisture loss. Because the Karl Fischer reaction is specific for the determination of water, other volatile constituents, which might be driven off by prolonged heating at high temperatures in the oven methods, are not calculated as water. The water is completely extracted because further treatment of the grain sample gives no increase in the amount of water in the solvent.

The Official Grain Standards of the United States specify an air-oven method for determining the moisture content of wheat, barley, oats, rye, rice, flaxseed and soybeans and a water-oven method for corn (7). Results of moisture determinations of these grains made by the official method were compared with results of determinations made with the

[2] The mention of a specific instrument is for the purpose of identification and does not imply endorsement by the U. S. Government.

Karl Fischer method (4). Results of the two methods agreed except in the cases of flaxseed, soybeans, corn and pea beans. In the determination of flaxseed and soybeans the official method was found to be in error as it determined volatile materials other than water. It was also shown that the official water-oven method used for corn and beans did not remove all the moisture.

The Karl Fischer method, shown to give accurate moisture content of seeds has some disadvantages. It requires a more skilled technician than the oven methods and takes more of his time. The Karl Fischer reagent, now improved and available commercially, is relatively expensive.

Because the errors of the oven methods are not found in the Karl Fischer method; because it eliminated the variation in apparent moisture content caused by drying to constant weight (an experiment will show how this variation occurs) the Karl Fischer, is used as a reference method in the present research.

EXPERIMENTAL PROCEDURE

The Relation Between Oven Drying Temperature and Apparent Moisture Content at Constant Weight

Portions of a thoroughly blended sample of hard red winter wheat, weighing about 10 grams, were placed in tared moisture boxes and weighed. All the boxes for each temperature to be tested were put in the vacuum oven at a mercury pressure of 5 mm or lower. The moisture content of the wheat was calculated after different time intervals by taking two boxes from the oven and determining their weight losses. This procedure was followed until continued heating gave no further weight loss. This was repeated for three different temperatures, 100°, 105°, and 110° C. The same procedure was followed with corn and flaxseed except that an air-oven was used. Corn samples were dried at 6 temperatures ranging from 94° to 105°. Flaxseed samples were dried at 5 temperatures ranging from 100° to 130°.

Comparison of the Air-oven Moisture Results from Ground Seeds Heated 1 Hour at 130° C. with Those from the Karl Fischer Method

Moisture determinations were made on 18 agricultural and vegetable seeds by the Karl Fischer method (4). Many different lots were used as sources for samples of each kind of seed tested. Chemical tests for completeness of extraction and interfering compounds were made for each kind of seed.

Other portions of the samples were ground on a Stein mill for 30 seconds. The Wiley mill was not used because the seeds gummed up the mill. Grinding in the Stein mill developed little or no heat. Since the grinding takes place in closed cup there is little danger of moisture loss during grinding. Three-gram portions of the ground seeds were placed in tared moisture boxes, weighed and heated for 1 hour at 130° C., as specified in the Official Grain Standards (7). Moisture contents were calculated from the weight losses.

Development of Oven Methods for Whole Seeds that Give Results Matching Those Obtained by the Karl Fischer Method

Moisture determinations were first made on the samples of 32 common agricultural seeds by the Karl Fischer method.

In selecting optimum temperatures at which to dry seeds in an air-oven, some seeds were found to require excessively long drying periods (over 300 hours) with temperatures less than 130° C. Other seeds, at a temperature of more than 100°, dried too quickly. Since many laboratories have only one air oven and since considerable time is often required to make the proper adjustments when the temperature setting of the oven is

changed, only two experimental temperatures, 100° and 130° were used. An exception was made in the case of flaxseed where the temperature used was 103°.[3]

Preliminary moisture determinations were made on a few samples of each kind of seed in a Brabender oven in which it was possible to weigh the samples at intervals without removing them from the oven. By making determinations on each kind of seed at the two temperatures, the time and temperature of oven drying giving results nearest to those obtained by the Karl Fischer method were found.

Using the selected times and temperatures final determinations of moisture content were made on the 32 kinds of seeds. Aluminum moisture dishes having a diameter of 55 mm and a depth of 15 mm were used. The amount of seed to be dried filled the dishes about half full. In the case of a few very light, bulky seeds, such as bluestem or bromegrass, the dishes were nearly full. Sample weights ranged from 4 to 10 grams. Samples were dried in a forced-draft oven. Previous experience with this oven in drying cereal grains had shown that it gave results equivalent to those obtained in the conventional gravity-convection air ovens.

RESULTS AND DISCUSSION

The Relation Between Oven-Drying Temperature and Apparent Moisture Content at Constant Weight

Some whole seeds dry to constant weight at different moisture levels. Duplicates of a sample of wheat (fig. 1) dried in a vacuum oven at 3 temperatures, ranging from 100° to 110° C., reached constant weight after about 175 hours at 3 apparent moisture contents ranging from 12.0 to 12.3 percent. Duplicates of a sample of corn (fig. 2) dried in an air oven at 6 temperatures, ranging from 94° to 105° reached constant weight after about 150 hours at 6 apparent moisture contents ranging from 10.5 to 11.4 percent. Duplicates of a sample of flaxseed (fig. 3) dried in an air oven at 5 temperatures, ranging from 100° to 130°, reached constant weight after from 20 to 60 hours at 5 apparent moisture contents ranging from 7.6 to 8.2 percent.

These variations in time of drying and apparent moisture content of whole seeds caused by the use of different temperatures of drying show the need for a standard procedure that will give accurate results. The difference in results also show that oven procedure should be standardized by comparing it with an accurate reference method that is independent of time and temperature.

Comparison of the Air-oven Moisture Results from Ground Seeds Heated 1 Hour at 130° C. with those from the Karl Fischer Method

Table 1 shows the difference in moisture content of 18 seeds when determined by this oven method and by the Karl Fischer method. The differences ranged from -0.27 to 2.00 percent. In 14 of the 18 seeds analyzed, the oven method gave high results. This variation from the results of the reference method shows that the oven method, using the same time and temperature for different seeds, does not give accurate results and indicates the need for standardization against a reference method.

Development of Oven Methods for Whole Seeds that Give Results Matching Those Obtained by the Karl Fischer Method

Air-oven procedures, using whole seeds, were standardized for 32 common agricultural seeds. For 23 of these seeds there has been no generally recognized or standardized method of determining moisture content.

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[3] Oven-moisture determinations were made on flaxseed by W. Haward Hunt, U. S. Department of Agriculture, Agricultural Marketing Service, Grain Division.

By comparison of results with the reference method, the chemical Karl Fischer method, a high temperature (130° C.) or a low temperature (100°) was selected for each kind of seed. (The flaxseed determinations were done at a different time and a temperature of 103° was used.) The time of drying for each seed was adjusted to give results matching those of the reference method.

Table 2 gives the times and temperatures selected for each seed and compares the results of the oven method with those of the Karl Fischer method. The average deviation of the moisture contents determined by the oven methods from those determined by the Karl Fischer method ranged from -0.14 to 0.10 percent. The standard deviations between the 2 methods ranged from 0.08 to 0.32 percent. Results of duplicate determinations by the oven methods agreed within 0.1 percent. Only 3 kinds of seeds required 20 hours or more for drying; only 3 required more than 10 hours, while 24 required 4 hours or less. Because the standardized methods use whole seed they save the time required for grinding.

Any laboratory assistant able to use an analytical balance should be able to determine the moisture content of whole seeds accurately by these standardized oven methods.

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APPENDIX

TABLE 1.--Comparison of the air-oven method (1 hour at 130° C.) with the Karl Fischer method for determining moisture in ground seeds

Kind of seed Common name	Botanical name	Moisture determined by K.F. method	Oven method	Moisture difference
	CRUCIFERAE:	*Percent*	*Percent*	*Percent*
Cabbage	Brassica oleracea var. capitata	8.17	7.90	- 0.27
Collard	Brassica oleracea var. acephala	8.39	8.18	- 0.21
Kale	Brassica oleracea var. acephala	5.70	5.92	+ 0.22
Mustard	Brassica juncea	6.52	6.71	+ 0.19
Rape	Brassica napus	6.72	6.48	- 0.24
Turnip	Brassica rapa	6.30	6.10	- 0.20
Radish	Raphanus sativus	5.38	5.83	+ 0.45
	GRAMINEAE:			
Bromegrass, smooth	Bromus inermis	11.11	11.26	+ 0.15
Orchardgrass	Dactylis glomerata	8.69	9.19	+ 0.50
Ryegrass, perennial	Lolium perenne	10.25	10.45	+ 0.20
Timothy	Phleum pratense	9.86	10.51	+ 0.65
Bluegrass, Kentucky	Poa pratensis	9.03	9.62	+ 0.59
	LEGUMINOSAE:			
Alfalfa	Medicago sativa	6.89	7.74	+ 0.85
Clover	Trifolium spp.	7.28	8.01	+ 0.73
	LILIACEAE:			
Onion	Allium cepa	7.67	8.55	+ 0.88
	UMBELLIFERAE:			
Carrot	Daucus carota	6.43	8.18	+ 1.75
Parsnip	Pastinaca sativa	5.73	5.83	+ 0.10
Parsley	Petroselinum hortense (P. crispum)	7.48	9.48	+ 2.00

TABLE 2.--Time and temperature of heating for oven methods for whole seeds which give results approximately equal to those obtained by the Karl Fischer method

Common name of seed	Botanical name of seed	Oven Method						
		No. of samples	Size of sample	Heating time		Tempera-ture	Average deviation from K.F. results	Standard deviation from K.F. results
	GRAMINEAE:		*Grams*	*Hrs.*	*Min.*	°C	*Percent*	*Percent*
Bentgrass, colonial	Agrostis tenuis	25	10	1	0	130	- 0.05	0.20
Bluestem, yellow	Andropogon ischaemum	2	1	1	0	100	+ 0.06	0.22
Oats (eastern origin)	Avena sativa	5	10	22	0	130	+ 0.03	0.13
Oats (Puget Sound origin)	Avena sativa	9	10	24	0	130	- 0.03	0.10
Bromegrass, smooth	Bromus inermis	22	4	0	50	130	- 0.02	0.19
Orchardgrass	Dactylis glomerata	23	5	1	0	130	+ 0.01	0.14
Fescue	Festuca spp.	24	5	3	0	130	0.00	0.14
Barley (eastern origin)	Hordeum vulgare	23	10	20	0	130	- 0.01	0.29
Ryegrass, perennial	Lolium perenne	4	5	3	0	130	- 0.11	0.14
Timothy	Phleum pratense	24	10	1	40	130	+ 0.02	0.14
Bluegrass, Kentucky	Poa pratensis	21	5	1	0	130	+ 0.02	0.15
Rye	Secale cereale	25	10	16	0	130	- 0.04	0.23
Sorghum	Sorghum vulgare	4	10	18	0	130	+ 0.05	0.17
Wheat	Triticum aestivum	72	10	19	0	130	+ 0.08	0.19

TABLE 2.--Time and Temperature of heating for oven methods for whole seeds which give results approximately equal to those obtained by the Karl Fischer method--Continued

Common name of seed	Botanical name of seed	Oven Method						
		No. of Samples	Size of sample	Heating time		Tempera-ture	Average deviation from K.F. results	Standard deviation from K.F. results
	LEGUMINOSAE:							
			Grams	*Hrs.*	*Min.*	°C.	*Percent*	*Percent*
Alfalfa	Medicago sativa	21	10	2	30	130	+ 0.02	0.11
Clover, alsike " crimson " white	Trifolium hybridum Trifolium incarnatum Trifolium repens	22	10	2	30	130	- 0.04	0.17
	LILIACEAE:							
Onion	Allium cepa	20	10	0	50	130	- 0.08	0.30
	LINACEAE:							
Flax	Linum usitatissimum	111	10	4	0	103	- 0.06	0.16
	UMBELLIFERAE:							
Carrot	Daucus carota	19	10	1	40	100	+ 0.05	0.17
Parsnip	Pastinaca sativa	1	10	1	0	100	- 0.10	0.10
Parsley	Petroselinum hortense	3	10	2	0	100	- 0.07	0.14
	COMPOSITAE:							
Safflower	Carthamus tinctorius	11	10	3	0	130	- 0.14	0.32
Sunflower	Helianthus annus	22	10	1	0	130	- 0.03	0.17

TABLE 2.--Time and temperature of heating for oven methods for whole seeds which give results approximately equal to those obtained by the Karl Fischer method--Continued

Common name of seed	Botanical name of seed	Oven Method					
		No. of samples	Size of samples	Heating time	Tempera-ture	Average deviation from K.F. results	Standard deviation from K.F. results
	CRUCIFERAE:		*Grams*	*Hrs. Min.*	°C	*Percent*	*Percent*
Cabbage	Brassica oleracea var. capitata	2	10	4 0	130	- 0.11	0.14
Collard	Brassica oleracea var. acephala	2	10	4 0	130	- 0.04	0.06
Kale	Brassica oleracea var. acephala	8	10	4 0	130	- 0.10	0.14
Mustard	Brassica juncea	20	10	4 0	130	- 0.02	0.32
Rape, annual	Brassica napus var. annua	2	10	4 0	130	- 0.08	0.08
Turnip	Brassica rapa	15	10	4 0	130	- 0.03	0.13
Radish	Raphanus sativus	30	10	1 10	130	+ 0.01	0.16

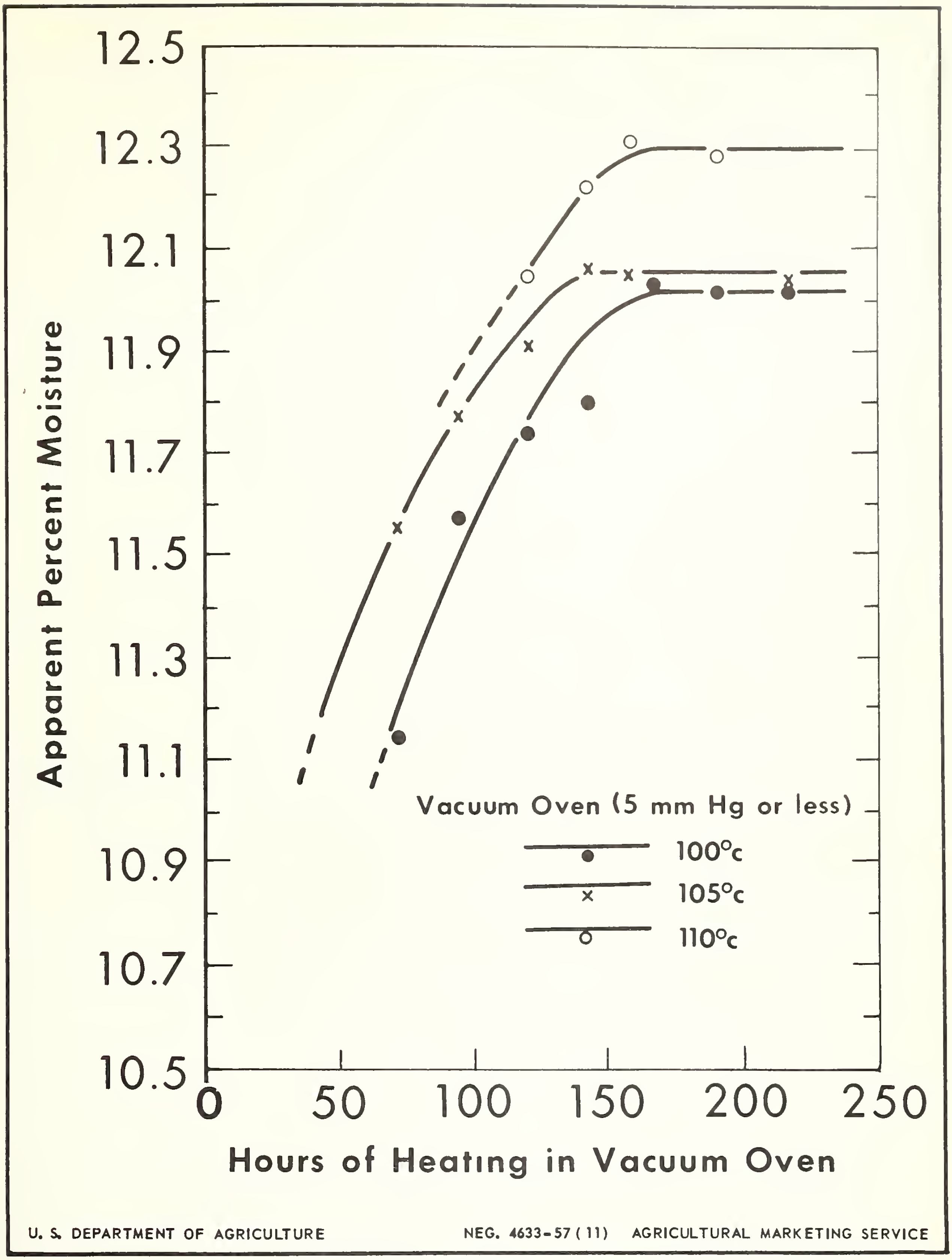

Figure 1.--Wheat (whole seed). Relation between oven drying temperature and apparent moisture content at constant weight.

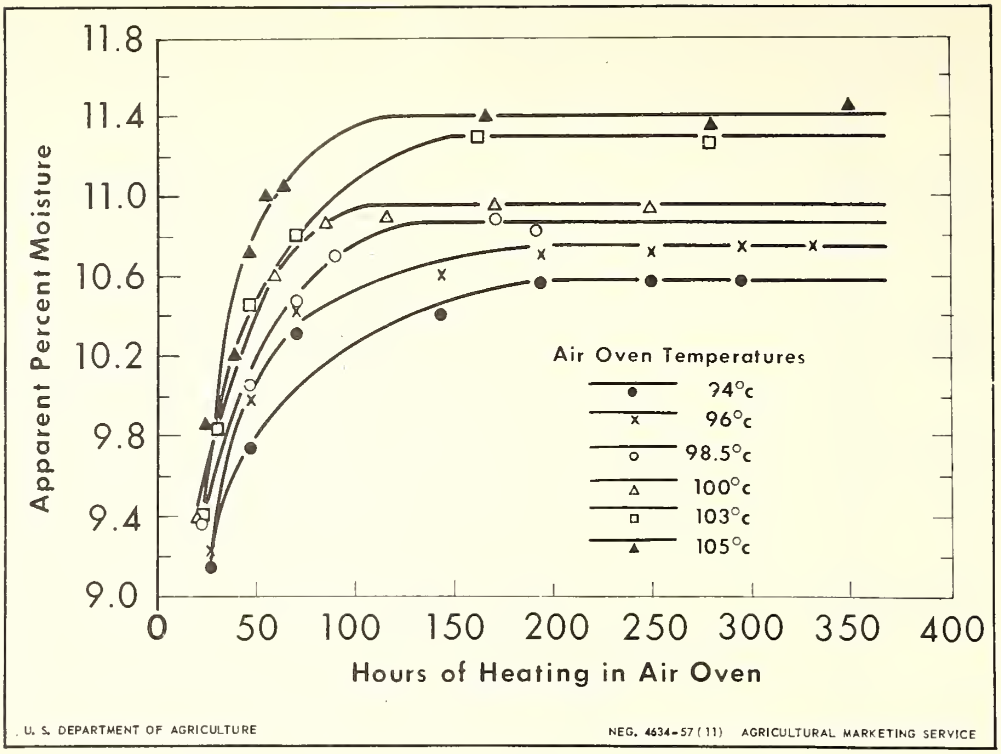

Figure 2.--Corn (whole seed). Relation between oven drying temperature and apparent moisture content at constant weight.

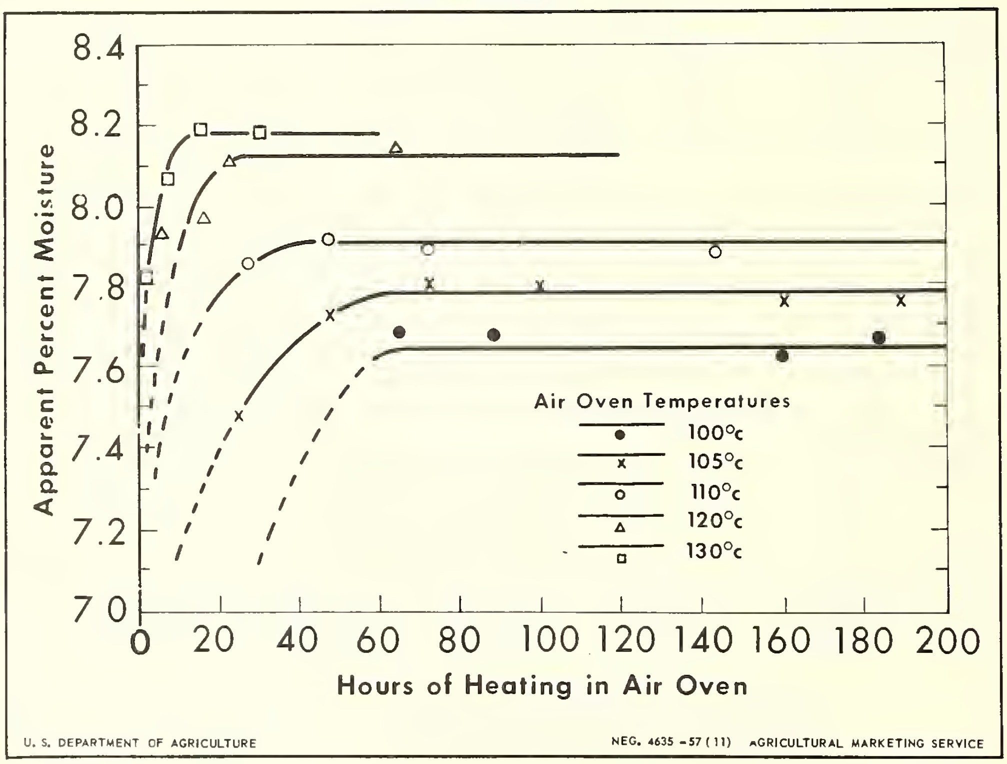

Figure 3.--Flaxseed (whole seed). Relation between oven drying temperature and apparent moisture content at constant weight.

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